

✓ NSWC TR 79–259





# PRODUCTION AND ASSESSMENT OF DAMAGED HIGH ENERGY PROPELLANT SAMPLES

BY WAYNE L. ELBAN
RESEARCH AND TECHNOLOGY DEPARTMENT

8 MAY 1980



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NSWC/TR-79-259/	145 HO44 726	
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Production and Assessment of Damag	, vod	
High Energy Propellant Samples	geu /	
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7 AUTHOR(s)		B. CONTRACT OR GRANT NUMBER(s)
Wall. Elban	ļ	SSP013
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Naval Surface Weapons Center		AREA & WORK UNIT NUMBERS
White Oak		0;
Silver Spring, Maryland 20910	'	
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17. DISTRIBUTION STATEMENT (of the abstract entered	in Block 20, if different from	n Report)
18 SUPPLEMENTARY NOTES		1
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19. KEY WORDS (Continue on reverse side if necessary ar	nd identify by block number)	
DDT Ultrasonio	cs	
Deformation Microscopy		
Fracture Thermal Si		1
Propellant Aquarium 1	Experiment	
20. ABSTRACT (Continue on reverse elde if necessary and	d identify by block number)	
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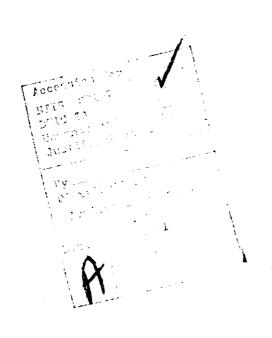
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sections of damaged and undamaged propellant, as well as on fracture surfaces when appropriate. Propellant recovered from a low amplitude shock sensitivity experiment, an aquarium shot, was also evaluated for comparison purposes.

In general, propellant samples that had been compressed at low strain rate did not undergo extensive damage. Crack densities were very low, and dewetting was the observed damage mechanism. Propellant samples exposed to liquid nitrogen exhibited numerous brittle fractures and little, if any, dewetting. Based on sound velocity measurements, the deformation was localized, leaving a large volume of material undisturbed. Propellant recovered from the aquarium experiment was damaged most extensively of all the material examined in this work. The crack number density and the area of crack surfaces/unit volume of propellant were highest. Sound velocity measurements indicate that dewetting did not occur in sizable portions of the material and that again the deformation was localized.



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#### **FOREWORD**

This work was performed for the High Energy Propellant Safety (HEPS) Program sponsored by the Strategic Systems Project Office under work request number N0003078WR82428 SSP013. The results and conclusions presented in this report concerning the production and characterization of damage in high energy propellants should be of interest to those working with the mechanical and fracture behavior of these materials. This work should also provide insight into some of the types of damage mechanisms than can occur to those investigating the influence damage has on propellant sensitivity and performance.

KURT F. MUELLER
By direction

#### **PREFACE**

R. R. Bernecker provided considerable insight into deflagration to detonation transition phenomena in high energy propellants and many suggestions that assisted in developing this work. The many helpful discussions on ultrasonic measurements with G. V. Blessing and A. L. Bertram are gratefully acknowledged. The microscopy was performed by M. K. Norr (scanning electron microscope) and R. H. Renner (light microscope). Several informative discussions were held with O. M. Dengel who demonstrated the feasibility of using exposure to liquid nitrogen to fracture high energy propellants. T. P. Liddiard provided the propellant recovered from the aquarium test and test information and characteristics. The numerous discussions with C. S. Coffey on heat generation due to deformation and fracture are recognized.

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#### I. INTRODUCTION

The most commonly accepted sequence of events describing how a high energy propellant rocket motor undergoes a transition from deflagration to detonation (DDT) begins with a ballistic malfunction which produces a multidimensional applied stress that heavily damages the propellant grain. Obviously, since the material is consumed, little is known about the nature of the damage (generated under these stress fields) which leads to DDT. Consequently, there is a need to develop laboratory techniques or small scale experiments for generating damaged propellant samples which possess varying amounts of cracking, porosity, and/or granularity. These samples would be available for a variety of sensitivity and performance testing including determination of shock sensitivity, closed tube DDT behavior, and augmented burning rate. It is also important to use (and develop where necessary) material characterization techniques to study the nature and extent of damage that occurs.

The deformation and fracture behavior of solid propellants has been the subject of considerable experimental and theoretical work. However, ultrasonics has been used sparingly as a tool to assess damage in propellants, and little effort has been devoted to microstructural analysis either during or after stressing. In a recent study, Knollman, Martinson, and Bellin<sup>2</sup> measured changes in longitudinal velocity and attenuation of ultrasonic waves as a discrete function of strain while the material (propellant or inert) was strained either in uniaxial tension, compression, or shear. It was concluded that the changes in wave speed and attenuation were caused by dewetting. In a separate study, Dreitzler and Shih<sup>3</sup> used ultrasonics to measure the as-manufactured porosity of propellants. A large decrease (almost 43%) in sound speed and a slight increase (about 7%) in attenuation were obtained when the porosity in the propellant increased from 0.35 to 1.35%.

Kincaid, J. F., "The Inadvertent Detonation of Large Solid Motors Loaded with High Energy Propellants," in ONR/AFOSR Workshop on Deflagration-to-Detonation Transition CPIA Publ. 299, 11-13 Jan 1978; publ 1978, pp. 5-18.

<sup>&</sup>lt;sup>2</sup>Knollman, G. C., Martinson, R. H., and Bellin, J. L., "Ultrasonic Assessment of Cumulative Internal Damage in Filled Polymers," <u>Journal of Applied Physics</u>, Vol. 50, No. 1, 1979, pp. 111-120.

Dreitzler, D. R. and Shih, C. C., "Porosity Measurement of Solid Propellants Using Ultrasonic Techniques," in Proceedings of the 1978 JANNAF Propulsion Meeting, Vol. I, CPIA Publ. 293, 14-16 Feb 1978; publ 1978, pp. 115-125.

The scanning electron microscope (SEM) has been used by Cornwell and Schapery<sup>4</sup> to observe in situ straining of solid propellant. A series of photomicrographs showing the material under increasing tensile strain was obtained; it was observed that cracks grow at or in the vicinity of the filler particle-polymer matrix interface. Large cavities around the filler particles were observed and appeared circular just prior to failure. No evidence was obtained for fracture occurring in filler particles.

The dynamic fracture and fragmentation behavior of high energy propellant at high strain rates has been investigated by Murri, Horie, and Curran<sup>5</sup> using a plate slap technique. From photomicrographs of the damaged propellant, it was hypothesized that HMX crystals are weakly bonded to the matrix and serve to nucleate cracks under applied tensile stresses. When the dynamic tensile stresses are sufficiently high, these cracks then grow and coalesce to form fragments. Microscopic examination of recovered fragments revealed that cracks were present and that debonding between the HMX and the propellant matrix had occurred. There was also evidence of fracture of HMX crystals.

This work was undertaken to subject a variety of high energy propellants to different stress fields and to assess the microstructural changes that occur. Low strain rate mechanical deformation in compression and low temperature thermal stressing were used to damage samples. These materials were tested nondestructively using ultrasonics in which longitudinal sound velocity measurements were made. Microscopic examination was performed on microtomed sections of damaged and undamaged propellant, as well as on fracture surfaces when appropriate. Propellant recovered from a low amplitude shock sensitivity experiment, an aquarium shot, was also evaluated for comparison purposes.

Cornwell, L. R. and Schapery, R. A., "SEM Study of the Microcracking in Strained Solid Propellant," Metallography, Vol. 8, No. 5, 1975, pp. 445-452.

Murri, W. J., Horie, Y., and Curran, D. R., "Fracture and Fragmentation of High Energy Propellant," SRI International Annual Report, Mar 1978.

#### II. EXPERIMENTAL

#### A. PRODUCTION OF DAMAGED SAMPLES

1. MECHANICAL DEFORMATION STUDIES. Four propellant formulations were utilized in this investigation. These were FKM (a modified double-base propellant), VOY-4 and VMX-2J, (modified double-base high energy propellants), and ALTU-16 (an experimental high energy composite propellant). All of these compositions contain ammonium perchlorate and HMX as crystalline oxidizers and aluminum as a fuel. A number of approximately 40 mm diameter, 13 mm high cylinders were machined from blocks of these materials. The machined surfaces were smooth, and the ends were parallel to each other to within 0.02 mm.

All samples were strained remotely in compression using a hydraulic press operating at a strain rate of approximately  $0.2 \, \mathrm{s}^{-1}$ . Engineering strains,

 $e = \frac{1-1}{1_0}$ , ranging from approximately -10 to -60% were achieved by pressing

to heights set by gage blocks for a dwell time of 120 s. Disks of 0.3 mm Teflon sheet were inserted between the sample and press platens to reduce friction in an effort to retain uniaxial strain at the higher strain levels and also as a safety precaution. Following the 120 s dwell time, the stress was completely released, and the samples were allowed to relax.

THERMAL STRESSING EXPERIMENTS. Only one propellant formulation, VRA-23, a modified double-base high energy propellant, was studied. This composition also contains ammonium perchlorate, HMX, and aluminum. Several approximately 38 mm diameter cylinders of varying heights (roughly ranging from 8 to 12 mm) were immersed in liquid nitrogen, as bare charges, for varying lengths of time up to 0.5 h. In addition, a series of experiments were performed in which samples, cut from 13 mm thick slab material, were exposed to less severe thermal gradients. Two approaches were pursued. First, one sample was placed in a Webber cold box (approximate temperature =  $-52^{\circ}$ C) for 0.5, 1, and 96 h. At the end of each exposure time interval, the sample was removed from the cold box, allowed to warm up to room temperature, and examined. The second approach involved surrounding the propellant with thermal insulation. Vermiculite was chosen as the insulating material. A new VRA-23 sample was placed in a small cardboard box surrounded by 3-4 mm of vermiculite (~ 1-2 mm granules), and the box was immersed in liquid nitrogen for 2 h. On removal, it was found that liquid nitrogen had permeated the box and was in contact with the sample. Again, the sample was allowed to warm up to room temperature and examined.

3. RECOVERED PROPELLANT FROM AQUARIUM SHOT. Some VRA-23 propellant that was recovered from an aquarium shot was examined many days after being shocked. The aquarium experiment is a shock sensitivity test  $^6$  for determining the threshold of burning and/or detonation for low amplitude-long duration shock waves. The configuration of the propellant sample had been two 50 X 50 X 13 mm thick slabs taped back to back which experienced a shock having an input pressure of approximately 1.25 GPa and a pulse duration of 25-30  $\mu$ s.

#### B. ASSESSMENT OF DAMAGE

Damage in propellant samples subjected to the various stress fields described previously was evaluated using ultrasonics and microscopy as well as visual observation. Residual strain values were calculated from axial dimensional measurements made before and after stressing.

A schematic of the ultrasonic apparatus used for making the axial longitudinal sound velocity measurements appears in Figure 1. The basic component is a broadband ultrasonic pulser/receiver (Panametrics Model 5055 PR) which is combined with two 25 mm diameter broadband contact transducers (Panametrics V102) having a center frequency of 1 MHz. Since the operation $^{7,8}$  of this instrumentation has been described in detail elsewhere, very little description will be given. This system can be operated in two different modes: pulse-echo and through-transmission. In pulse-echo, ultrasonic pulses are received by the transmitting transducer after being reflected off the backwall, while in through-transmission, a separate receiving transducer is positioned at the backwall. The latter mode of operation was employed when the strained samples were too highly attenuating to permit pulse-echo operation. A viscous couplant, such as honey or silicone stopcock grease, is used to transmit the ultrasonic energy between the transducer and the test specimen. Signal amplitude versus time output is displayed on an oscilloscope (Tektronix Model 545B), and transit time measurements (horizontal axis) are made directly from the cathode ray tube screen. It is estimated that the velocities obtained for the system operating in pulse-echo are accurate to about 1%, while through- transmission errors may be as high as 5%.

The SEM was used extensively to examine microtomed\* sections of stressed propellant samples. These were compared to microtomed surfaces of unstressed material in an effort to identify the various damage mechanisms that were operating.

<sup>&</sup>lt;sup>6</sup>Liddiard, T. P., "The Initiation of Burning in High Explosives by Shock Waves," in <u>Proceedings of the Fourth Symposium on Detonation</u>, Publ. ACR-126, 12-15 Oct 1965; publ 1966, pp. 487-495.

Krautkramer, J. and Krautkramer, H., <u>Ultrasonic Testing of Materials</u> (New York: Springer-Verlag, 1969), pp. 151-196.

<sup>&</sup>lt;sup>8</sup>Pollard, H. F., <u>Sound Waves in Solids</u> (London: Pion Ltd., 1977), pp. 157-159.

<sup>\*</sup>Some sectioning was performed using a single-edge razor instead of a microtome.

No difference in appearance could be detected in viewing surfaces of unstressed material obtained with these two cutting tools. Consequently, sectioned propellant will always be referred to as "microtomed".

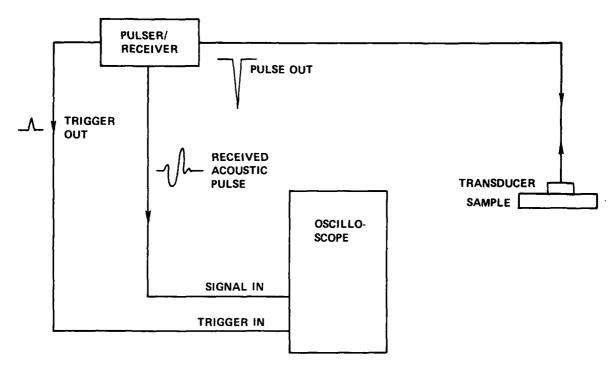


FIGURE 1 SCHEMATIC DIAGRAM OF ULTRASONIC APPARATUS (PULSE-ECHO OPERATION)

(TAKEN FROM REFERENCE 11).

In addition, many of the fracture surfaces that resulted were examined using the SEM. The operation and use of the SEM<sup>9</sup> has been discussed in detail and will not be reviewed in this work. Sample preparation is worth mentioning, however. All of the propellant specimens that were examined were non-conductive and required the application of a thin conducting coating. This was accomplished by vacuum depositing first a carbon layer, followed by a layer of a gold-palladium alloy. In an effort to minimize electron beam damage, samples were viewed at low magnification (25-50X) first, and then the magnification was increased to 1000X maximum. There was no evidence of beam damage in any of the samples at these magnifications during the exposure periods.

Some of the microtomed specimens were also examined using the light microscope operating at low magnification (about 73X). Although optical microscopy is an exceedingly valuable technique for investigating structures of materials, 10 it does have a serious deficiency that hinders its application in this work. Usually, the depth of field is limited sufficiently to prohibit viewing the entire specimen in focus, even though microtoming provides a reasonably flat surface. Although some worthwhile photomicrographs were obtained with the light microscope, those chosen for discussion purposes were obtained using the SEM.

Hearle, J. W. S., Sparrow, J. T., and Cross, P. M., The Use of the Scanning Electron Microscope (Oxford: Pergamon Press, 1972).

<sup>10</sup> Richardson, J. H., Optical Microscopy for the Materials Sciences (New York: Marcel Dekker, Inc., 1971).

#### III. RESULTS AND DISCUSSION

#### A. MECHANICAL DEFORMATION STUDIES

The results 11 for straining FKM, VOY-4, VMX-2J, and ALTU-16 in compression have been reported previously, and only a summary will be given, including some of the more interesting results. In general, the samples that had been mechanically deformed at the low strain rate used in this work did not exhibit extensive damage. Crack densities were found to be very low. The propellant exhibiting the most visible damage was FKM, and the cylinders subjected to the three highest strains appear in Figure 2. For this particular material, there were numerous cracks (see samples strained to -58.5 and -69.8%) at the casting grain boundaries. Very little cracking was visible in the cylinders of the remaining compositions. Longitudinal velocity measurements 11 one hour after compressing to various strain levels are given in Figure 3 for the various propellants. In each instance, there was a decrease in sound speed with increasing applied strain, although the rate of decrease differed markedly. Reductions in sound speed, by as much as 88%, were obtained for ALTU-16.

Microsopic examination of microtomed cross-sections of stressed propellant did not reveal gross cracking in the propellant (except for FKM) or cracking in ingredient particles to be widespread damage mechanisms. It is considered unlikely that these make a significant contribution to the change in longitudinal velocity as a function of compressive strain. The damage mechanism that was observed is dewetting 12 or failure of the interfacial bond between filler particles and the polymer matrix. As an example, a set of SEM photomicrographs 11 that were obtained for sections of ALTU-16 is given Figure 4. Each sample exhibited numerous debonded crystals, but no incident cracks were observed. There was some evidence of cracked crystals that is attributed to the microtoming operation. Although it is difficult to distinguish between the extent of damage for the surfaces of samples strained to various levels, the sample strained to -58.8% did display the most debonding. It was concluded that vacuole formation (see Appendix A) is responsible (either partially or totally) for the decrease in longitudinal velocity with increasing strain. 11 It was not determined

Elban, W. L., Bertram, A. L., Blessing, G. V., and Bernecker, R. R., "The Use of Ultrasonics for the Nondestructive Evaluation of Damage in Mechanically Deformed Propellants," Unclassified; unlimited report within Proceedings of the 15<sup>th</sup> JANNAF Combustion Meeting, Vol. III, CPIA Publ. 297, 11-15 Sep 1978; publ 1979 (CONFIDENTIAL), pp. 49-73.

<sup>12</sup> Oberth, A. E., "Principle of Strength Reinforcement in Filler Rubbers," Rubber Chemistry and Technology, Vol. 40, No. 5, 1967, pp. 1337-1363.

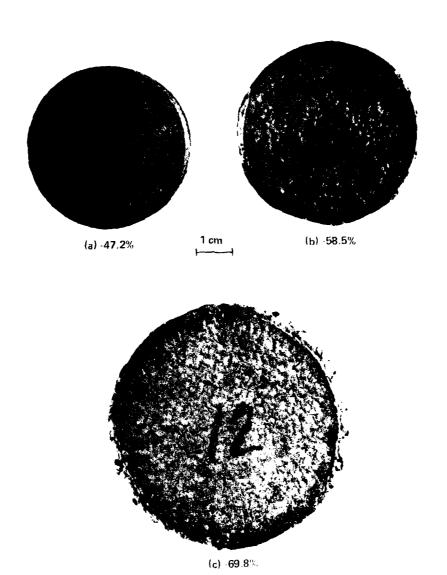


FIGURE 2 CYLINDRICAL CHARGES (41 mm DIA INITIALLY) OF FKM STRAINED IN COMPRESSION TO (a) -47.2%, (b) -58.5%, AND (c) -69.8%

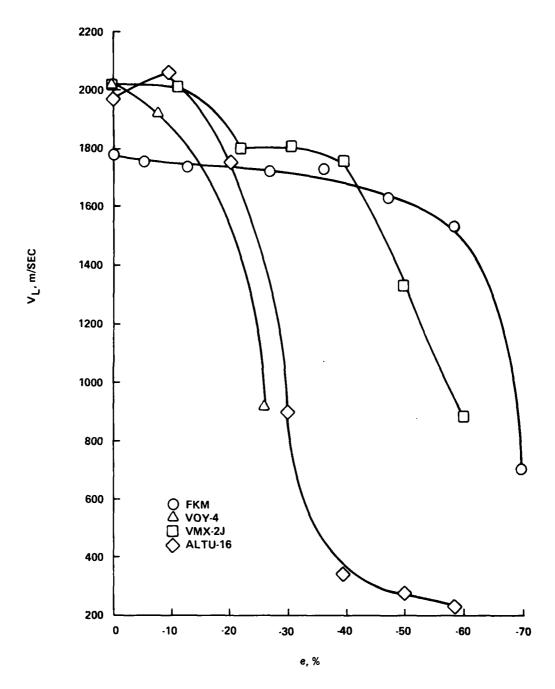


FIGURE 3 LONGITUDINAL VELOCITY ONE HOUR AFTER COMPRESSING VERSUS APPLIED ENGINEERING COMPRESSIVE STRAIN FOR PROPELLANT SAMPLES (NOMINAL 40 mm DIA x 13 mm HIGH)

(TAKEN FROM REFERENCE II).

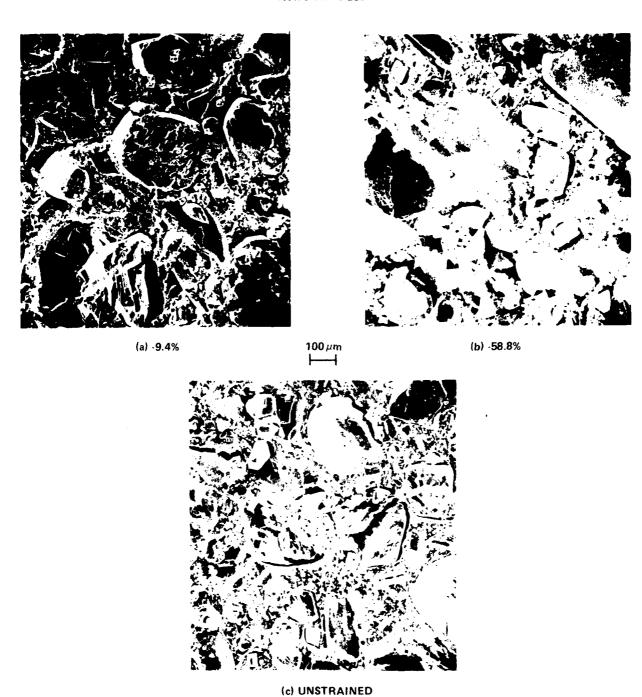


FIGURE 4 SEM PHOTOMICROGRAPHS OF MICROTOMED SECTIONS OF ALTU-16
(a) STRAINED TO -9.4%, (b) STRAINED TO -58.8%, AND (c) UNSTRAINED
(TAKEN FROM REFERENCE II).

whether non-visible damage, such as deformation in the propellant binder or dislocation generation in the filler crystals, also makes a significant contribution.

#### B. THERMAL STRESSING EXPERIMENTS

The results obtained for bare cylindrical charges (38 mm diameter) of VRA-23 immersed in liquid nitrogen for various lengths of time are given in Table 1. Each of the samples cracked into a number of pieces (see bottom two cylinders in Figure 5), and numerous small cracks were visible in each of the pieces. Visual inspection of the fracture surfaces revealed that the material underwent brittle fracture. The length of each sample was measured at room temperature before and after quenching, and no dimensional change was detected within the accuracy of the measurement. Only a slight decrease in longitudinal velocity was obtained for a sample immersed in liquid nitrogen for thirty minutes and allowed to warm to room temperature.

A number of interesting observations can be made from the SEM photomicrographs taken of material that had been immersed in liquid nitrogen. For comparison purposes, a microtomed cross-section of undamaged VRA-23 appears in Figure 6. The surface that results is monolithic in appearance with the largest exposed crystals (probably HMX) being about 200 µm. In a few instances, it appears that the cutting operation has caused debonding to occur at the binder-crystal interface. The fracture surface that results when a bare cylindrical charge of VRA-23 is immersed in liquid nitrogen is shown in Figure 7. Although this surface reveals more topographical detail than the microtomed surface, it is still fairly flat and featureless. Additional cracks do not intersect this primary fracture surface, and there is little indication that dewetting has occurred. However, numerous pores having characteristic dimensions ranging from 5 to 15 µm can be observed. The origin of these pores is not definitely known, but it is felt some water soluble ingredient (probably ammonium perchlorate) has been leached from the surface while the sample warmed to room temperature. It was observed that almost immediately after removal from liquid nitrogen, the sample fragments became covered by ice which eventually melted and ran off.

A microtomed cross-section approximately 1 mm behind the primary fracture surface generated during immersion in liquid nitrogen is displayed in Figure 8. Several cracks that did not extend to an exterior surface are visible. These cracks are straight and have smooth surfaces, exhibiting a largely brittle character. The porosity noted in the fracture surface displayed in Figure 7 was not observed in this cross-section, indicating that this porosity is a surface phenomena that does not occur in the bulk material. Perhaps the most interesting feature in Figure 8 (see bottom portion of lower magnification photomicrograph) is the crystal (believed to be HMX) that was fractured as a crack propagated through the propellant. Clearly, for these sample loading conditions, the binderparticle interfacial bond exceeded the tensile fracture stress of the HMX crystal. Evidence for fracture of HMX crystals was also obtained from examining recovered VRA-23 propellant that had experienced high strain rate impact by a flyer plate.<sup>5</sup> However, the propellant fracture surfaces that resulted in these experiments do not reveal nearly as much brittle character as those obtained in the present work. It is concluded that the fracture obtained by bare charge immersion in liquid nitrogen was too severe to be useful in subsequent DDT experiments or comparison sensitivity tests.

See footnote 5 on page 8.

TABLE 1 COMPILATION OF RESULTS OBTAINED FOR BARE CYLINDRICAL (38MM DIA) CHARGES OF VRA-23 IMMERSED IN LIQUID NITROGEN

ν Λ (π/s)	1850	Not Determined	Not Determined
Fracture	Many Pieces; Brittle	Many Pieces; Brittle	Many Pieces; Brittle
Audible Cracking Noise	< 1 min	~35 s	~20 s
Immersion Time	30 min	~36 s	~21 s
Final Sample Length (mm)	11.62- 11.68	9.68- 9.70	8.25- 8.28
Initial Sample Length	11.65- 11.68	9.70- 9.72	8.28- 8.30

\*Pulse-echo;  $f_c$  = 1 MHz;  $V_L$  for undamaged VRA-23 is 1880 m/s.

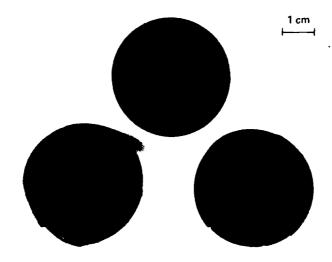


FIGURE 5 CYLINDRICAL CHARGES (38 mm DIA) OF VRA-23
TOP: CONTROL, HAS NOT BEEN THERMALLY
STRESSED. BOTTOM: PIECES RECOVERED FROM
BARE CHARGES IMMERSED IN LIQUID NITROGEN.

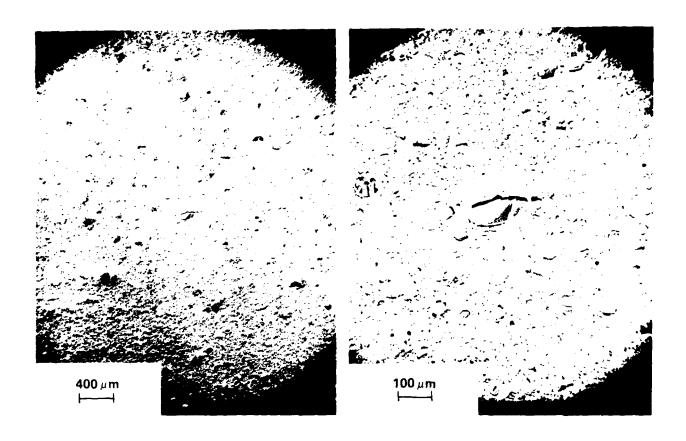


FIGURE 6 SEM PHOTOMICROGRAPHS OF MICROTOMED SECTION OF UNSTRAINED VRA-23

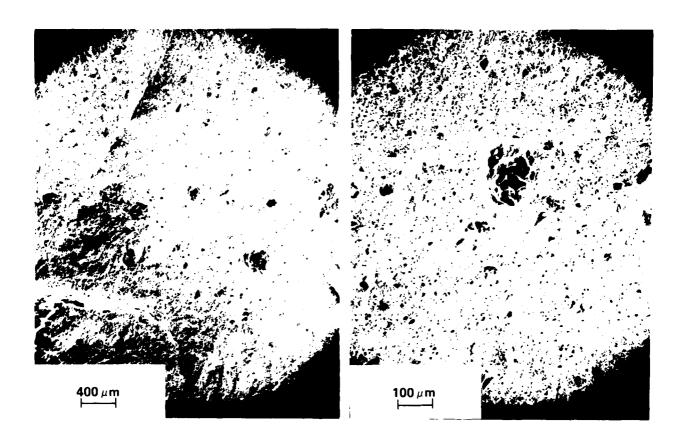


FIGURE 7 SEM PHOTOMICROGRAPHS OF FRACTURE SURFACE OF VRA-23 THAT RESULTED WHEN BARE CHARGE WAS IMMERSED IN LIQUID NITROGEN

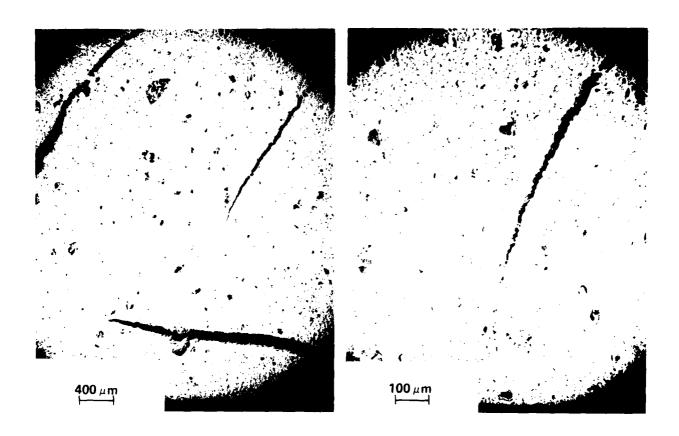


FIGURE 8 SEM PHOTOMICROGRAPHS OF MICROTOMED SECTION APPROXIMATELY
1 mm BEHIND PRIMARY FRACTURE SURFACE OF VRA-23 THAT RESULTED
FROM LIQUID NITROGEN QUENCHING

In an attempt to lessen the severity of the cracking, a series of experiments were performed in which samples of VRA-23 (taken from 13 mm thick slab material) were exposed to smaller thermal gradients. These results are compiled in Table 2. Virtually no change in longitudinal velocity was obtained for either the sample exposed to -52°C for 96 h or the sample insulated with vermiculite and immersed in liquid nitrogen. It is concluded that these smaller temperature gradient experiments were not successful in providing material having high crack densities (in fact, no cracks were observed). Although it should be possible to develop a controlled thermal stressing technique for obtaining samples having large densities of smaller length cracks, the nature of brittle fracture is such to make this a very difficult task. According to the Griffith energy-balance concept13 for characterizing crack propagation in purely brittle solids, if the applied stress exceeds a critical level, the crack will propagate spontaneously without limit. Thus, it is unlikely that during thermal stressing, cracks will propagate in brittle materials without many extending to exterior surfaces, leaving the material in a fragmented state.

In comparing the results from the thermal stressing experiments with the findings from the mechanical deformation studies, it is clear that the crack number density and the area of crack surface/unit volume of propellant increased significantly for the material exposed to liquid nitrogen. The low temperature brittle fracture indicates a significant reduction in ultimate elongation and points to the temperature dependency of the mechanical properties of VRA-23. This is expected since the stress-strain behavior of materials is temperature and strain rate dependent and since strong strain rate sensitivity for VRA-23 has been demonstrated from split Hopkinson bar experiments. The constancy of the sound speed measurements (see Table 1) suggests that very little dewetting occurred in the samples that were exposed to liquid nitrogen. Confirmed by microscopic examination, this indicates that the deformation leading to fracture is highly localized, leaving much of the material virtually undeformed.

#### C. RECOVERED PROPELLANT FROM AQUARIUM SHOT

The VRA-23 propellant recovered from the aquarium experiment was heavily fragmented into granules and pieces of various sizes. The two largest pieces appear in Figure 9. Visual inspection of this material revealed that extensive cracking had occurred. The cracks exhibited considerable tortuosity and were unlike the straight faceted cracks associated with thermal stressing, as seen in Figure 5. The exposed fracture surfaces were rough indicating that the cracking was non-brittle. Residual strain and longitudinal sound velocity measurements (in the thickness dimension) were made on perimeter and center regions of the larger recovered piece. These results are listed in Table 3. Each region had a small compressive residual strain, while the longitudinal velocity was found to increase slightly. An increase in longitudinal sound velocity was observed similarly (see Figure 3) for VMX-2J and ALTU-16 having small compressive residual strains obtained by mechanical stressing. It is important

Lawn, B. R. and Wilshaw, T. R., Fracture of Brittle Solids (Cambridge: Cambridge University Press, 1975), pp. 5-9.

<sup>&</sup>lt;sup>5</sup>See footnote 5 on page 8.

 $<sup>^{11}\</sup>mathrm{See}$  footnote 11 on page 13.

COMPILATION OF RESULTS OBTAINED FOR VRA-23 CHARGES SUBJECTED TO SMALLER TEMPERATURE GRADIENTS TABLE 2

Observations	No Cracking	No Cracking	No Cracking	No Cracking
V <sub>L</sub> (m/s)	Not Determined	Not Determined	1890	1870
Exposure Time (h)	0.5	-	96	2
Temperature (oC)	-51	-52	-52	Liquid Nitrosen **

\*Pulse-echo; f = 1 MHz;  $v_{\rm L}$  for undamaged VRA-23 is 1880 m/s.

Liquid Nitrogen \*\*

\*\*Sample insulated with about 3-4 mm of vermiculite.

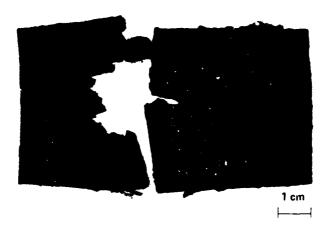


FIGURE 9 LARGEST PIECES (INITIALLY 50 x 50 mm) OF VRA-23 RECOVERED FROM AQUARIUM SHOT

TABLE 3 COMPILATION OF RESULTS OBTAINED FOR VRA-23 CHARGES RECOVERED FROM AQUARIUM SHOT

Observations	Extensive Cracking; Non-Brittle	Extensive Cracking; Non-Brittle
V. (m/s)	1940	1910
Residual Strain	-1.2	9.0-
Sample Location	Perimeter	Center

\*Through-transmission;  $f_c$  = 1 MHz;  $V_L$  for undamaged VRA-23 is 1880 m/s.

to note that the residual strain and longitudinal velocity did not change significantly for material that had undergone considerable cracking.

Some SEM photomicrographs were obtained, allowing several additional interesting observations. An exposed fracture surface is displayed in Figure 10. Considerable roughness is evident along with numerous secondary cracks intersecting the primary fracture surface. Interfacial bond failures are visible in a few instances, although there is a lack of discernible filler particles. Light gray secondary phase particles are also conspicuous, particularly in the higher magnification photomicrograph. The origin of these particles is not known. They may be reaction products from an early stage burning reaction (see SEM photomicrographs of burned propellant surfaces that were extinguished obtained by Derr and Boggs) 14 or corrosion products resulting from the fractured material being exposed to the aqueous environment. Additional attempts to locate secondary phase particles on exposed fracture surfaces from other regions of the samples were unsuccessful.

A microtomed cross-section approximately 1 mm behind the primary fracture surface appears in Figure 11. The tortuous character of the cracks is quite visible, but no secondary phase particles could be found. Further, there was no other indication that a burning reaction had occurred in or near the cracked regions. The fracture surface of a non-exposed "zero-width" crack is shown in Figure 12. This surface closely resembled the one appearing in Figure 10, except for the absence of secondary phase particles. Thus, it appears that the presence of the secondary phase particles is highly localized. In addition, no fractured HMX crystals were observed in any of the photomicrographs taken of propellant recovered from the aquarium test.

Of all of the samples examined in this work, the damage was most extensive in the propellant recovered from the aquarium shot. The small compressive residual strain and the slight increase in longitudinal velocity suggest that the samples experienced a small applied compressive stress and that dewetting did not occur. Apparently, there was increased intimacy at the binder-filler interface, and yet considerable tearing and rupture occurred. This is difficult to resolve considering the sequence of events leading to failure in composite solid propellants given in Appendix A. The most likely explanation lies in the fact that, for the aquarium experiment, the deformation occurs at high strain rate (estimated to be greater than 104 s-1). In addition, the strain rate sensitivity of the material is probably such that the elongation at break is very low for the loading conditions of this experiment. Thus, the possibility for dewetting to occur is virtually eliminated. Analogous to propellant exposed to liquid nitrogen, the deformation leading to fracture in propellant recovered from the aquarium shot must be localized also, leaving sizable portions of relatively undisturbed material for the sound velocity measurements that were obtained. The failure pattern observed in Figure 9 is not consistent with any of the conventional compression failure modes 15:

Derr, R. L. and Boggs, T. L., "Role of Scanning Electron Microscopy in the Study of Solid Propellant Combustion: Part III The Surface Structure and Profile Characteristics of Burning Composite Solid Propellants," Combustion Science and Technology, Vol. 1, No. 5, 1970, pp. 369-384.

Kendall, K., "Complexities of Compression Failure," Proceedings of the Royal Society of London, Ser. A, Vol. 361, No. 1705, 1978, pp. 245-263.

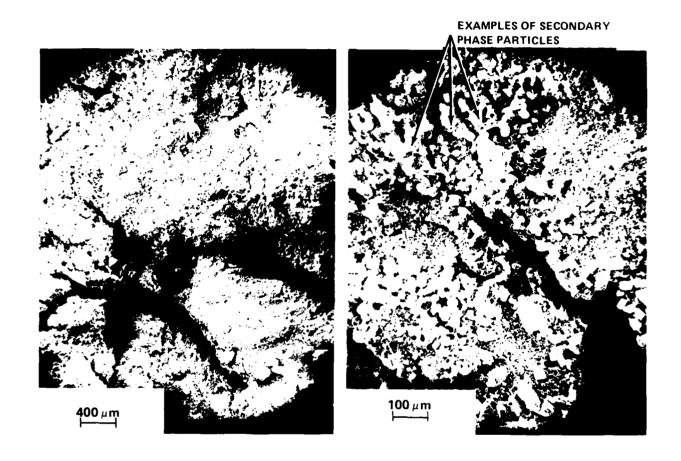


FIGURE 10 SEM PHOTOMICROGRAPHS OF EXPOSED FRACTURE SURFACE OF VRA-23 RECOVERED FROM AQUARIUM SHOT

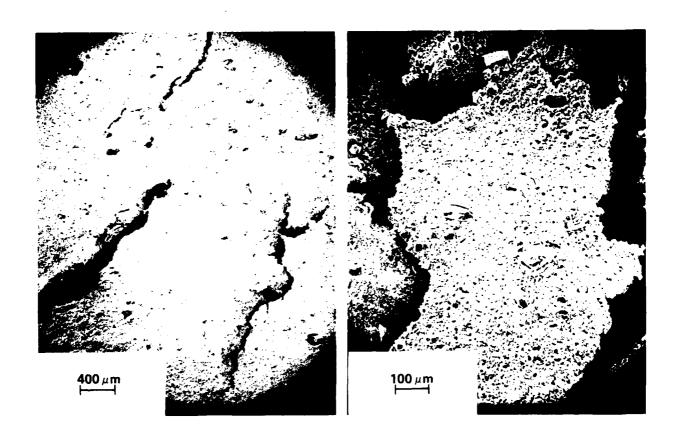


FIGURE 11 SEM PHOTOMICROGRAPHS OF MICROTOMED SECTION APPROXIMATELY
1 mm BEHIND PRIMARY FRACTURE SURFACE OF VRA-23 RECOVERED
FROM AQUARIUM SHOT

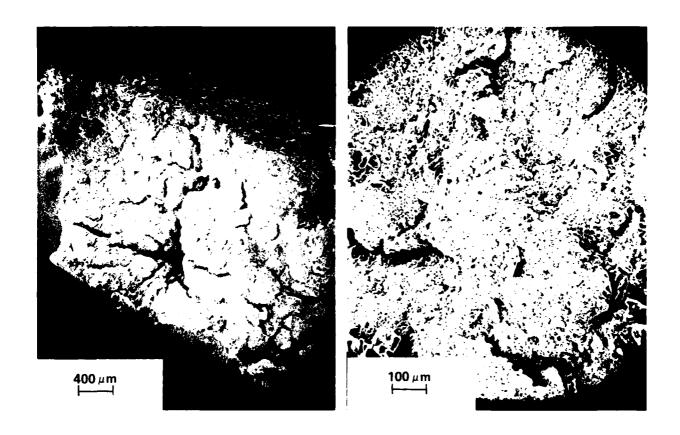


FIGURE 12 SEM PHOTOMICROGRAPHS OF NON-EXPOSED FRACTURE SURFACE OF VRA-23 RECOVERED FROM AQUARIUM SHOT

plastic yielding, cone failure, and axial splitting. The reason for this is not readily understood. Fracture caused by out-gassing of reaction products from an early stage shock-induced burning reaction in the bulk propellant is one possible explanation. Damage associated with postshock impact may have occurred also.

The extensive damage found in propellant recovered from the aquarium experiment serves to emphasize the attractiveness of developing an inexpensive variation of the aquarium test that would use a low amplitude-long duration shock to damage propellant for subsequent sensitivity and performance testing. However, this does not involve an in situ arrangement where damage and testing occur simultaneously (or follow very closely in sequence). This is an important consideration since there is increasing evidence that considerable localized heating occurs when materials undergo deformation and fracture, expecially at high strain rates  $(\ge 10^3~{\rm s}^{-1})$ . Winter and Field<sup>16</sup> investigated the impact initiation of explosives by striking single crystal targets (explosive) with tiny spherical particles (inert). Initiation resulted when critical conditions for particle size and velocity were exceeded. A nice feature of this experiment is the ability to examine and characterize the deformation that resulted when the impact conditions were slightly below the critical conditions necessary for initiation. Typically, the impact sites were smooth plastic indentations and few fractures were found. It was observed further that the deformation was highly localized in narrow bands of material, which were proposed to form by adiabatic shear. Although it was suggested that the mechanism responsible for initiation is the production of high local temperatures as a result of the adiabatic shear deformation, no temperature measurements were attempted.

However, in somewhat related work the temperature rise at the tip of a fast-moving crack has been found to be quite high for several different types of materials. Fuller, Fox, and Field 17 used thermocouples, a temperature sensitive liquid crystal, and an infrared detector to obtain temperature rises of about 400 and 500°K for polystyrene and polymethylmethacrylate, respectively. The temperature at the tip of cracks in glass and quartz was measured by Weichert and Schönert 18 using a four wavelength radiation thermometer and interpreting the observed light emission as thermal radiation. Values of 3200 and 4700°K were obtained for glass and quartz respectively. Thus, it is clear that the high energy density in the vicinity of a fast moving crack causes irreversible deformation which is dissipated as heat. There is a need to develop laboratory experiments to determine the contribution this heat production has on the DDT susceptibility of high energy propellants.

Winter, R. E. and Field, J. E., "The Role of Localized Plastic Flow in the Impact Initiation of Explosives," Proceedings of the Royal Society of London, Ser. A, Vol. 343, No. 1634, 1975, pp. 399-413.

<sup>&</sup>lt;sup>17</sup>Fuller, K. N. G., Fox, P. G., and Field, J. E., "The Temperature Rise at the Tip of Fast-Moving Cracks in Glassy Polymers," <u>Proceedings of the Royal Society of London</u>, Ser. A, Vol. 341, No. 1627, 1975, pp. 537-557.

Weichert, R. and Schönert, K., "Heat Generation at the Tip of a Moving Crack,"

Journal of the Mechanics and Physics of Solids, Vol. 26, No. 3, 1978, pp. 151-161.

#### IV. SUMMARY AND CONCLUSIONS

- A. In general, propellant samples that had been compressed at low strain rate did not undergo extensive damage. Crack densities were very low, and dewetting was the observed damage mechanism.
- B. Propellant samples exposed to liquid nitrogen exhibited numerous brittle fractures and little, if any, dewetting. Based on sound velocity measurements, the deformation was localized, leaving a large volume of material undisturbed. The low temperature brittleness indicates a substantial reduction in ultimate elongation.
- C. Propellant recovered from the aquarium experiment was damaged most extensively of all the material examined in this work. The crack number density and the area of crack surfaces/unit volume of propellant were highest. Although the recovered material was heavily fragmented into granules and pieces of various size, sound velocity measurements indicate that dewetting did not occur in sizable portions of the material and that the deformation was localized.

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# VI. ABBREVIATIONS, ACRONYMS, AND SYMBOLS

v <sub>L</sub>	longitudinal sound velocity	m/s
е	engineering strain	%
<b>L</b>	final sample length	mm
l <sub>o</sub>	initial sample length	mm
f	center frequency	MHz

#### APPENDIX A

#### MODEL OF THE FAILURE PROCESS IN COMPOSITE SOLID PROPELLANTS

Tensile failure in composite solid propellants is considered to occur in a multiple step process. Shimizu and Tanemura<sup>19</sup> list the following sequence of events (except at very low temperatures):

- (1) Underwetting process elastic deformation before the initiation of separation between the binder-filler interface.
- (2) Dewetting process initiation and growth of vacuoles.
- (3) Tearing process crack initiating and growing to be catastrophically large crack.
- (4) Rupture process after the start of abrupt macro-rupture from one of the catastrophically large cracks.

Although the above is strictly for tensile failure, similar processes are envisioned for composite propellants subjected to compressive stresses.

Shimizu, M. and Tanemura, T., "Fracture Energy of a Composite Solid Propellant," in Proceedings of the Mechanical Behavior of Materials Symposium, Vol. I, 21-24 Aug 1974; publ 1974, pp. 589-598.

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